TEMPORAL INFLUENCES OF SEASONAL HYPOXIA ON SEDIMENT BIOGEOCHEMISTRY IN COASTAL SEDIMENTS

A Thesis

by

KAREN S. SELL

Submitted to the Office of Graduate Studies of Texas A&M University in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

August 2003

Major Subject: Oceanography
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Approved as to style and content by:

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August 2003

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ABSTRACT

Temporal Influences of Seasonal Hypoxia on Sediment Biogeochemistry in Coastal Sediments. (August 2003)

Karen S. Sell, B.S., Eckerd College

Chair of Committee: Dr. John W. Morse

Bottom water hypoxia and its influence on the environment have been topics of increasing concern for many coastal regions. This research addresses both spatial and temporal variability in sediment biogeochemistry at the southeastern region of Corpus Christi Bay, TX, where seasonal (summer) hypoxia occurs. Traditional techniques for determination of a variety of dissolved and solid components, benthic oxygen demand, and sulfate reduction rates were augmented by measurements using solid state microelectrodes to simultaneously determine concentrations of dissolved O$_2$, Mn$^{2+}$, Fe$^{2+}$, and ΣH$_2$S in multiple small - interval (1 mm) depth profiles of sediment microcosms. Oxygen concentrations in the overlying water were manipulated in the sediment microcosms and electrode depth profile measurements were made over ~ 500 hours of experimentation. Laboratory and field microelectrode results were in good agreement for both norm - oxic and anoxic time periods. Results indicated that iron (Fe$^{2+}$) and sulfide (ΣH$_2$S) were the redox reactive species in these sediments. During hypoxic conditions an upward migration of dissolved Fe$^{2+}$ and ΣH$_2$S through the sediment column and, at times, into the overlying water was observed as the dissolved oxygen
concentrations decreased. A corresponding decline in the vertical extent of these redox species occurred when the overlying water was re-oxidized. When both dissolved iron and sulfide coexisted, FeS minerals were formed in the sediment, preventing sulfide diffusion into the overlying water. However, after a long duration of hypoxia (> 200 hours) this buffering capacity was exceeded and both iron and sulfide penetrated into the overlying waters. Results indicated that iron may have a greater influence on hypoxia than sulfide because its concentration in the overlying waters during induced hypoxia was an order of magnitude greater than those of sulfide. Moreover, in the southeastern region of the Bay, where mixing was minimal and the water column was shallow, the sediments alone may have caused the onset of the hypoxic event in a relatively short time period (< 5.5 days). These results demonstrated that in shallow marine environments where seasonal hypoxia occurs, such as Corpus Christi Bay, the associated major changes that take place in the sediment biogeochemistry must be included in benthic - pelagic models for overlying water hypoxia.
DEDICATION

This work is dedicated to God as the One who has given me this scientific gift, the endurance to finish, and the dream to go on. To my parents, grandparents, and family who have supported me through the way. To my best-friend and partner, Eric, who has given me companionship, foundation, and balance. To Jana & Mimi Ann Watts who have helped guide me to my new life in Christ and who have become my family away from home. To my cat “son” Sammy, who is always there for me to cuddle. To the United States of America troops who fight so I can study in the land of the free, now and forever, without the threat of terror.
ACKNOWLEDGEMENTS

I thank Dr. John Morse for his support and guidance through my 3 years as his student, my committee for their thoughts and review, and the Scherck Chair and fellowship for contributions to fund this work and my studies. I thank Andy Hebert, who bestowed his electrode knowledge on to me and helped in field collections, without him, this work would not have been accomplished. Thanks to Megan Singer, who as a student worker helped make electrodes, organize materials, and run selected samples. To Melanie Beazley and Dwight Geldhill for their support inside and outside of the laboratory. To Dr. Ken Dunton, Dr. Paul Montagna, Dr. Wayne Gardner, Mark McCarthy, all at UTMSI who arranged for lab space, housing, boat and equipment use, and extra experimentation. Thanks to Craig Aumack for his diving expertise and assistance in the field. Thanks to Dr. Gill Rowe & the DGOMBE project for Gyre use. To GERG for the use of the Whaler for five weeks. Finally, thanks to Eddie Webb and Sheridon Haye, whose engineering expertise allowed for the analyses in this work to occur with ease and speed.
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CHAPTER I
INTRODUCTION

Hypoxia Background

Hypoxia is traditionally defined when dissolved oxygen is < 2 mg O₂ L⁻¹ (62.5 µM) in the water column. Hypoxic conditions commonly occur in bottom waters because of the organic material decomposition at the sediment surface. After deposition of particulate organic matter to the benthos from the entire water column (detritus, fecal pellets, etc.), aerobic bacteria decompose the organic material and consume the available oxygen, resulting in hypoxia or even anoxia (absence of oxygen), where the anaerobic metabolism dominates. One of the casual mechanisms for hypoxia is stratification of the water column where available surface water oxygen is prevented from reaching the bottom waters, this can be intensified by the lack of wind/tidal mixing, increased temperatures, and fresh (nutrient rich) water runoff. Hypoxia may be viewed as an ecological stressor because very few benthic animals are able to tolerate the physiological stress associated with extended exposure to low oxygen conditions (Jørgenson, 1980; Harper et al., 1981; Ritter and Montagna, 1999). During seasonal hypoxia, some direct effects of low oxygen include reduced benthic abundance, biomass, and diversity, avoidance by mobile fauna, emergence of infauna, and even death (Harper et al., 1981; Dauer and Ranasinghe, 1992; Buzzelli et al., 2002; Morehead et al., 2002).

The format follows Estuaries.
Some areas suffer from benthic faunal extinction during the hypoxic event and then recolonization following the event (Jørgenson, 1980). Hypoxia can even affect higher trophic levels by decreasing the available prey, where commercial and recreational fishery stocks may be disturbed.

Hypoxia has been studied in at least 44 reported places in the world (Morehead et al., 2002) where studies on seasonal hypoxia include but are not limited to Limfjorden, Denmark (Jørgenson, 1980), Corpus Christi Bay (Ritter and Montagna, 1999), west of the Mississippi River Delta (Harper et al., 1981; Rabalais et al., 1994; Morse and Rowe, 1999; Justic et al., 1995; and others), Chesapeake Bay (Dauer and Ranasinghe, 1992; Sagasti et al., 2001), Neuse River Estuary, North Carolina (Buzzelli et al., 2002), and the Baltic Sea (Gamenick et al., 1996). Even though hypoxia may be a natural phenomenon, events are likely intensified by anthropogenic activities, where a major impact on it is increased temperatures due to global warming (Morehead et al., 2002). Further, eutrophication caused from the use of fertilizers (nitrogen and phosphorous) can increase primary production on the surface, where there would otherwise be limited production, inducing bottom water hypoxia from the increased downward flux of organic material (Turner and Rabalais, 1991). Hypoxia is a global problem where the understanding of the sources, effects, and processes that contribute to it are of interest to all environmental scientists and managers alike.

The phenomenon of hypoxia in bottom waters has been a topic of scientific interest for many decades; however the traditional focus has been strictly on the biological response, and not the dynamics of biogeochemical processes that occur during
such episodes. Previous studies of the benthic biogeochemical processes in seasonally hypoxic areas recommend more research to better understand regional processes (e.g. Morse and Rowe, 1999). The purpose of this study was to quantify sediment biogeochemical parameters that are affected during a hypoxic event, enabling a better understanding of the impact of the sediment on the surrounding marine environment during subsequent changes in overlying water oxygen concentrations.

**Redox Chemistry**

During hypoxia and anoxia, anaerobic metabolism dominates and the degradation of organic matter occurs with the most energetically favorable reduction pathway depending on the available terminal electron acceptor (respiration reactions Table 1.1). The bacterial reactions that were chosen for measurement in this study were oxygen, nitrate, manganese, iron, and sulfate reduction (detection limits in methods section). The most frequently detected dissolved components in this study were sulfide and iron (II) via microelectrodes. Fe^{2+} is a product of the pathway of iron reduction. In a typical sediment profile it occurred at the shallow sediment depths (< 2.5 cm), most prominently at high concentrations (> 1000 µM), and within a narrow band. This steep gradient and narrow peak may indicate intense metal reduction-oxidation cycles (Kristiansen et al., 2002). H$_2$S is produced by sulfate reduction. It generally occurred deeper (~ 3 – 10 cm) in the sediment column than iron in lower concentrations (< 250 µM) and wider (several cm) bands (Figure 1.1).
TABLE 1.1. Measured organic matter decomposition reactions and corresponding free energy states (Berner, 1980).

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<td>-475</td>
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<tr>
<td>(CH$<em>2$O)$</em>{106}$(NH$<em>3$)$</em>{16}$(H$_3$PO$_4$) + 138O$_2$ → 106CO$_2$ + 16HNO + H$_3$PO$_4$ + 122H$_2$O</td>
<td></td>
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<td>Nitrate reduction:</td>
<td>-448</td>
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<td>(CH$<em>2$O)$</em>{106}$(NH$<em>3$)$</em>{16}$(H$_3$PO$_4$) + 94.4HNO$_3$ → 106CO$_2$ + 55.2N$_2$ + H$_3$PO$_4$ + 177.2H$_2$O</td>
<td></td>
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<td>Manganese reduction:</td>
<td>-349</td>
</tr>
<tr>
<td>(CH$<em>2$O)$</em>{106}$(NH$<em>3$)$</em>{16}$(H$_3$PO$_4$) + 236MnO$_2$ → 236Mn$^{2+}$ + 106CO$_2$ + N$_2$ + H$_3$PO$_4$ + 177.2H$_2$O</td>
<td></td>
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<tr>
<td>Iron reduction:</td>
<td>-114</td>
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<tr>
<td>(CH$<em>2$O)$</em>{106}$(NH$<em>3$)$</em>{16}$(H$_3$PO$_4$) + 212Fe$_2$O$_3$ + 848H$^+$ → 424Fe$^{2+}$ + 106CO$_2$ + 16NH$_3$ + H$_3$PO$_4$ + 53OH$_2$0</td>
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<tr>
<td>Sulfate reduction:</td>
<td>-77</td>
</tr>
<tr>
<td>(CH$<em>2$O)$</em>{106}$(NH$<em>3$)$</em>{16}$(H$_3$PO$_4$) + 53SO$_4^{2-}$ → 106CO$_2$ + 16NH$_3$ + H$_3$PO$_4$ + 53S$^{2-}$ + 106H$_2$O</td>
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Fig. 1.1. A typical profile of reduced iron and sulfide in studied sediments (0 mm is the sediment surface).
Sediment Chemistry

The solid phase chemistry of iron and sulfide are important in benthic sedimentary biogeochemistry. Iron sulfide minerals are primarily the products of bacterially mediated sulfate reduction processes that occur rapidly in near surficial sediments. Iron sulfide minerals that are found in most recent sediments include the potential acid volatile sulfide (AVS) minerals, amorphous sulfide (FeS), mackinawite (FeS$_{0.9}$), and greigite (Fe$_3$S$_4$). Pyrite (FeS$_2$) is the most stable (net) and usually by far the most abundant iron sulfide mineral. Iron is a common component in most sediment and can exist in many chemical forms (i.e. clays, iron oxides, oxyhydroxides). Iron reactivity can vary but generally iron oxides / oxyhydroxides are the most reactive form (Canfield, 1986). Sulfur is the other major component of iron sulfide minerals and its primary source in sediments is from dissolved sulfate. The average concentration of dissolved sulfate in seawater is 28 mM at a salinity of 35, but total reactive sulfides (TRS) can exceed the equivalent of several pore volumes of seawater due to transport and diffusion. Sulfate reduction is the principle terminal microbial process in anoxic sediment where sulfate is available (Capone and Kiene, 1988).

In porewater, the Fe$^{2+}$ and H$_2$S concentrations can be governed by the solubility of mackinawite. When dissolved Fe$^{2+}$ and ΣH$_2$S are present and detectable, the saturation state of the porewaters with respect to this mineral can be determined (Equations 1-8). The activity coefficients for Fe$^{2+}$ are based on Davison (1979) and for H$^+$, and HS$^-$ are based on values from Millero and Schreiber (1982).
\[ \alpha_{\text{Fe}^{2+}} = \gamma_{\text{Fe}^{2+}} \cdot m_{\text{Fe}^{2+}} \]  

(1)

\[ \alpha_{\text{H}^+} = \gamma_{\text{H}^+} \cdot m_{\text{H}^+} \]  

(2)

\[ \sum H_2S = m_{H_2S} + m_{HS^-} \]  

(3)

\[ K_1 = \frac{(\alpha_{HS^-} \cdot \alpha_{H^+})}{\alpha_{H_2S}} = 10^{-6.9} \]  

(Khodakovskii et. al., 1965)  

(4)

\[ K_{\text{FeS(mack)}} = \frac{\alpha_{HS^-} \cdot \alpha_{\text{Fe}^{2+}}}{\alpha_{H^+}} \]  

(5)

\[ K_{\text{FeS(mack)}} = 10^{-2.96} \]  

(Morse et al., 1987)  

(6)

\[ m_{HS^-} = \frac{K_1 \gamma_{H_2S} \sum H_2S}{(\gamma_{HS^-} \cdot \alpha_{H^+} + K_1 \cdot \gamma_{H_2S})} \]  

(7)

\[ \Omega = \frac{\alpha_{HS^-} \cdot \alpha_{\text{Fe}^{2+}}}{K_{\text{FeS(mack)}} \cdot \alpha_{H^+}} \]  

(8)

Where, \( \alpha \) is the activity product, \( \gamma \) is the activity coefficient, \( m \) is the molality, \( K \) is the solubility product for mackinawite, \( K_1 \) is the dissociation constant for sulfide, and \( \Omega \) is the saturation state with respect to mackinawite. By using the literature values of the activity coefficients for \( \text{Fe}^{2+} \) (0.47) and \( \text{H}^+ \) (0.85), \( \text{Fe}^{2+} \) porewater concentrations from microelectrode measurements, and assuming a pH of 7.4, the activity products for \( \text{Fe}^{2+} \) and \( \text{H}^+ \) can be calculated (Eq. 1-2). The molality of \( \sum H_2S \) can be calculated by using an activity coefficient for \( \text{HS}^- \) of 0.79 (Eq. 3). Using the literature values for the
dissociation constant (Eq. 4), solubility product (Eq. 5 - 6), and the microelectrode measurements for porewater $\sum H_2S$ and assuming an activity coefficient for hydrogen sulfide of 1, equation 7 can be solved and in turn, the saturation state with respect to mackinawite can also be calculated (Eq. 8).

A wide range of saturation states have been observed for various iron sulfide minerals within the same sediment and therefore it is difficult to put specific generalizations on the controls of the formation of these minerals (Morse et al., 1987). Even so, the equilibrium between the dissolved and solid phases can be a major factor in the uptake and release of iron and sulfide. Usually > 98% of the sulfide is oxidized, but under hypoxic / anoxic conditions sulfide can accumulate in both the porewaters and potentially more importantly in the solid phase as iron sulfides. Iron sulfide pools normally act as a buffer binding the excesses of sulfide (and iron) as FeS and FeS$_2$, preventing them from leaching into the overlying waters (Jørgenson, 1980). However, if reactive iron is no longer available to bind with the sulfide, then this buffer is no longer present and most of the sulfides will exclusively be in the porewater phase, where then diffusion into the overlying waters is possible.

**Diffusion**

During hypoxia, the major mode of transport of ions changes from bioturbation to diffusion (Morse and Rowe, 1999). Molecular diffusion is the net motion of matter resulting from the random motion of ions, molecules, or atoms. Diffusion in the porewaters of sediments can be described best by Fick’s First and Second Laws of
Diffusion (Berner, 1980). The first law (Equation 8) describes diffusion across a plane where the second law (Equations 9 - 11) describes the rate of change at depth x (Crank, 1975).

Fick’s First Law:

\[ J = -D_s \]  

(9)

Fick’s Second Law:

\[ \frac{\partial C}{\partial t} = D_s \frac{\partial^2 C}{\partial x^2} + \left( \frac{\partial D_s}{\partial x} + \frac{D_s \partial \phi}{\phi \partial x} \right) \frac{\partial C}{\partial x} , \]  

(10)

\( C \) = Concentration of component
\( D_s \) = Diffusion coefficient for ion in sediment
\( \phi \) = Porosity

The above can be simplified if D is constant to,

\[ \frac{\partial C}{\partial t} \bigg|_x = D_s \frac{\partial^2 C}{\partial x^2} \]  

(11)

During a hypoxic event, the release of Fe^{2+} and, in intense conditions of depleted oxygen, H_{2}S into the bottom waters causes extensive damage to the benthic flora and fauna. The surrounding organisms are poisoned by the sulfide through diffusion into their skin. The main toxic effect to metazoans is the blocking of oxidative enzymes
which ultimately cause them to suffocate (Theirmann et al., 2000). Some metazoans have adapted to sulfide conditions by creating an anaerobic capacity, while other like the nematode, *Oncholaimus campylocercoides*, may possess sulfide oxidation capabilities (Theirmann et al., 2000). However, overall, these are opportunistic species and most other species do not retain abilities to survive in long – term (days - weeks) hypoxic conditions.

The diffusion of H$_2$S and Fe$^{2+}$ can occur into the overlying waters during induced hypoxic studies. This is likely due to the depleted oxygen conditions, during which oxidation processes may be unable to keep up with the supply of Fe$^{2+}$ from below, leading to a release into the overlying water and eventual exhaustion of the pools (Kristiansen et al., 2002). If this occurs, sulfide may ultimately be released, since Fe pools may act as buffers, delaying the release of H$_2$S. Figure 1.2 demonstrates iron release into the overlying waters. Sulfide, although present at 1 mm below the sediment – water interface, was not released in this case. This was probably due to the formation of mackinawite and other iron-sulfide minerals where the two constituents coexisted. Once these dissolved species have been released into the overlying waters they may help drive bottom water hypoxia, where subsequent re – oxidation will occur at the pycnocline, retarding bottom-water re - aeration, thereby helping to maintain hypoxic / anoxic conditions (Tuttle et al, 1987).
Fig. 1.2. Iron and sulfide depth profiles from a measured core in this study during overlying water hypoxia. To the right the coexistence of the two reduced species is accentuated.
Texas Coastal Hypoxia

Most Texas bays are shallow (typically ~1 - 2 m) with consistent wind, storm events, and tidal flushing that promote mixing and exchange with coastal waters. Based on shallow waters and wind induced vertical mixing, Texas bays do not seem vulnerable to hypoxic episodes. However, hypoxic events have been reported to occur in several locales seasonally. The well known areas include Corpus Christi Bay, Offatts Bayou, and along the upper Texas coastline (Rowe et al., 2002, Ritter and Montagna, 1999; Cooper and Morse, 1996; Rabalais et al., 1994). All of these hypoxic events are seasonal, although each has different physical mechanisms as their causes. However, the fundamental casual mechanism that they all share is stratification of the water column, inducing bottom water hypoxia. Stratification prevents the surface oxygen from vertically mixing to the bottom waters and at the bottom where the benthic respiration is utilizing the available oxygen in the process of degrading organic matter, oxygen if not re-supplied can be quickly consumed. Further contributors such as increased temperatures during the summer months can increase respiration through the Q_{10} effect and increased nutrient loads from river runoff also deplete oxygen levels by enhancing surface primary production and the supply of organic material to the bottom sediments. Considerable research has been done to determine the causes of hypoxia in these regions and its effect on the associated fauna and biota. However, much less effort has been made to understand the sediment biogeochemistry associated with such events (Krom and Burner, 1980; Roden and Tuttle, 1992; Cooper and Morse, 1996; Morse and Rowe, 1999; Kristiansen et al., 2002; Rowe et al., 2002).
Research Objectives

The purpose of this thesis was to develop an experimental technique that makes possible the manipulation of oxygen in a sediment microcosm to mimic that of an episodic and short-lived hypoxic event and to determine which redox species (H$_2$S, Fe$^{2+}$, Mn$^{2+}$) played a dominate role in the stability of the chemical environment. The overall goal was to monitor the chemical behavior of the sediment and porewaters and to assess the impact of the sediment biogeochemistry on the system during hypoxia.

The hypotheses that were tested are:

n$_0$ = The manipulation (decrease / increase) of overlying water O$_2$ concentrations will cause an associated (upward / downward) migration of reduced iron and sulfide.

n$_1$ = Laboratory microcosm results will agree with field measurements.

n$_2$ = During induced experimental and field hypoxia, reduced iron and sulfide will be released into the overlying waters.

n$_3$ = The formation of FeS minerals will prevent sulfide from leaching into the overlying waters.

n$_4$ = Dissolved Fe$^{2+}$ and ΣH$_2$S will be sufficient to delay recovery from the hypoxic event.
CHAPTER II
LABORATORY METHOD DEVELOPMENT

Introduction

Although hypoxia has been studied extensively, the emphasis of most studies has been primarily on the causes and resulting organism responses. Little research has been conducted on the characteristics and dynamics of sediment biogeochemistry associated with hypoxic events. This lack of prior experimental studies presented the challenge of developing laboratory methods that would be effective, efficient, and repeatable. In order to develop appropriate study methods, a sediment microcosm, 16 cm in diameter with overlying water, was taken from Redfish Bay, TX, because of its easy accessibility and soft bottom type. The associated redox changes in the porewaters of the microcosm core were measured, with solid state microelectrodes, during overlying water oxygen concentration manipulation. Since this type of porewater monitoring had not previously been accomplished (to the author’s knowledge) the technique could prove useful to understanding the biogeochemical response to episodic hypoxia. Thus, the technique developed was subsequently used for analyses at known seasonally hypoxic locations, which are described in the following chapters of this thesis.
Methodology

Study Location

RedFish Bay, located northeast of Corpus Christi Bay, was small and shallow (<3 m) with many seagrass communities. This bay was conveniently located close to the University of Texas Marine Science Institute in Port Aransas, TX, where all field work was conducted (Fig. 2.1). The area was visited in the spring of 2002 and hypoxia was known to occur in this region, but sulfide production had been previously recorded (Morse, unpublished).

Sample Collection

Sediment cores were pushed - cored by hand, while free diving, with care to minimize disturbance during the collection activities. Cores were collected in ~ 1 m of water depth and were promptly capped using clasped fitted tops, and immediately taken to the laboratory for further analyses. Three 16 cm diameter, 40 cm - long Plexiglas incubation cores for microelectrode measurements and laboratory oxygen manipulation were taken within 1 m of each other.

Microelectrode Measurements

Concentrations of O₂, Mn²⁺, Fe²⁺, and ΣH₂S in the pore waters of the top 10 cm of sediment and 10 mm of overlying bottom waters were measured by anodic stripping voltammetry, using solid state potentiometric microelectrodes (Luther et al., 1998; Brendel, 1995). “Voltammetry is the application of a potential ramp with the subsequent
Fig. 2.1. Satellite image of Corpus Christi Bay and Redfish Bay.
measurement of current, when a chemical species reacts at the electrode” (Brendel, 1995). The electroanalytical techniques used in this study were linear sweep and cyclic voltammetry which was applied to the potential range from -0.1 V to 2.1 V. Solid state microelectrodes with small tip diameters (100 µm) have many advantages over other electrodes with large tip diameters (refer to Brendel, 1995 for further inquiry). An advantage to using microelectrodes over traditional methods is that microelectrodes are a non-destructive sampling technique where the measurement of dissolved concentrations can be obtained on a finer scale than generally possible with other techniques, providing a detailed survey of the geochemical events that occur in sediment porewaters through time during the experimentally induced hypoxia. Electrodes were made in the laboratory using a hollow glass rod in which a cable was soldered onto a 100 µm gold wire tip and epoxied inside the glass rod. The tip of the electrodes were polished with sandpaper and diamond paste, plated with mercury nitrate, and then polarized in NaOH solution. The plating technique allowed for a mercury coating over the gold wire while the polarization allowed the Hg to diffuse into the Au tip providing lower minimum detection limits and a more durable amalgam (Brendel, 1995). Minimum detection limits for O₂, Mn²⁺, Fe²⁺ and H₂S were 5 µM, 5 µM, 15 µM, and 0.2 µM, respectively (Brendel, 1995). Calibration of every electrode was based on the pilot ion method where Mn was the standardized ion. Manganese was chosen as the pilot ion because of its slower oxidation kinetics compared to that of reduced iron and sulfide. A known volume of seawater (35 ppt.) was put into a cell and sparged with nitrogen, then serial additions of 0.036 M MnCl₂ was added, while an electrode scan at each
concentration was made. Stirring during electrode scanning was stopped to lower the
noise in the signal. The temperature of the cell was not regulated but the influence of
average room temperature (20 – 25°C) on the Mn slope (0.7) was priorly determined to
be less than 0.059 (Brendal, 1995). After determination of the slope for manganese, the
slope for O₂, H₂S, and Fe²⁺ was possible because of their known empirical slope ratios
with respect to Mn²⁺ (Brendel, 1995). This ratio can be different depending on each
electrode, however, as long as the ratio has been determined, the concentration of the
measured constituent can be calculated. Furthermore, electrodes were only used in this
work if their manganese slope was comparable (+/- 0.2) to the previous work of Brendal
(slope = 0.7).

The system included a laptop computer with Labview© software to display
millivolts (-0.1 v – -2.8 v) vs. amperage readings for all measured species. The
computer was connected to an Analytical Instrument Systems (AIS) DLK (model 100A)
voltammetric analyzer to which the microelectrodes were also connected (Figure 2.2).
After measurements were taken, peak heights were determined automatically through a
specifically engineered Labview© program where a second derivative baseline fit was
made on the voltammogram, following closely to Peakfit© software. Millimeter scale
depth resolution intervals were obtained using a micromanipulator, where small
microenvironments were represented and measured.

The sediment cores for microelectrode analyses were sealed and aerated with a
simple aquarium air pump until the initial electrode measurements were made. Aeration
was processed through a hydration flask to prevent evaporation of overlying water. The
Fig. 2.2. Picture of the laboratory experimental set-up including computer, DLK 100A, fish tank, micromanipulator, aerator, and some cores (stirring magnet, not applicable).
vertical (i.e., downcore) measurement steps were every 1 mm for the first 10 mm and then every 2 mm down to 60 mm, then in steps of 5 mm until 100 mm was reached. Upon completion of the initial survey, aeration was stopped, and the overlying water became hypoxic in the sealed cores. Re-aeration of the core was performed when dissolved redox reactive species were detectable in the overlying water and then aeration was terminated once again when the core had reached normoxic conditions in the overlying water (> 200 µM O₂) to induce a “natural” and “short-lived” hypoxic setting. Electrode profiles were made throughout the entirety of the experiment. All profiles were made in different locations in the core to avoid potential error produced by previous electrode penetration and associated small scale core disturbance. Unfortunately, attempted stirring techniques, including a magnetic and a flow through system were deemed unfavorable for this design because there were a large number of cores to be stirred at once. Therefore, all reported fluxes are to be considered minimum values for the experiments of induced hypoxia.

**Results**

**Visual Observations**

Striations of colors in the sediment were observed to increase as the core experienced hypoxia. Changes from light brown, to red, to grey, to black occurred, with scattered black spots, all occurred simultaneously (Figure 2.3). With time, the sediment
colors became more pronounced and micro-zones of iron oxides and sulfide minerals appeared to be present. Bioturbation was not visually noticeable (e.g. macrofauna).

**Microelectrode Results**

Total dissolved sulfide (ΣH₂S) had by far the highest concentration of the dissolved reduced porewater components measured (0 - 70 µM). The experimental method was therefore evaluated by studying the laboratory reaction of this species. The microelectrode profile results in Figure 2.4 (a) show ΣH₂S initially occurred deep (60-80 mm) downcore. As the time of induced hypoxia increased, ΣH₂S was observed at more shallow depths in the core, until it eventually diffused into the overlying water column after 166 hours of oxygen depletion. When the core was re-oxidized, ΣH₂S was again observed to occur deeper in the core but at higher concentrations than initially measured. Figure 2.4 (b), obtained by using Matlab© software, shows this behavior for the top 60 mm of sediment, where the darker regions represent low sulfide concentrations. To further illustrate the core chemical response during the induced hypoxia, the ΣH₂S concentrations at 2 mm and at 85 mm were graphed with experiment time in Figure 2.5. When the air supply was turned off, sulfide concentrations increased at both depths, but the 2 mm ΣH₂S increased more dramatically. When the air supply was turned back on, the 85 mm ΣH₂S continued to increase (3 – fold) but the 2 mm sulfide decreased to below detection limits.
Fig. 2.3. Photographs of a cross-section of laboratory cores during reduced oxygen conditions. The pictures show the many areas of sediment micro-heterogeneity within a core.
Fig. 2.4. a). Sulfide profiles with experiment time b). Sulfide concentration variations with experiment time.

Fig. 2.5. The sulfide concentration of the top 2 mm and the bottom 85 mm of sediment with time.
Discussion

The results of this study demonstrated that induced hypoxia caused the upward migration of the initial depth at which observed dissolved sulfide was measured in the incubated sediment microcosm. With further hypoxia, the concentration of sulfide increased throughout the zone of active sulfate reduction. This may have occurred from sulfide evolving upward from below and/or a decrease in sulfide oxidation allowed its concentration to increase at shallow core depths (Jørgenson, 1980). When, the system was re-oxidized, the upper porewaters with dissolved sulfide were oxidized. However, a similar decrease in sulfide was not observed deeper in the core where it continued to increase (3 - fold). A cause for this could be decreased bioturbation due to organism death from the hypoxic conditions, allowing less air to reach the deeper sediment porewaters, creating a rich environment for sulfate reducers until the rate of oxygen diffusion allowed aeration at these depths to occur. H₂S accumulation from below the measured depth may also contribute to this increase. At these deeper sediment depths, the H₂S was therefore “banked” in the sediment where it was protected from oxidation and was stored. The upper 50 – 70 mm of the sediment porewaters displayed the most chemical variability and should therefore be the focus of measurement.

Conclusions

This experimental study demonstrated that the sediment redox chemistry undergoes major changes in response to variations in the overlying water oxygen content over a range of tens of centimeters. Surface sediment and lower sediment layers showed
different responses during re-oxidation and evidence for the “banking” of redox species was seen in the lower sediments. This may be an important phenomena in future hypoxic studies since the “banking” of reduced species can prolong hypoxia. The illustrated laboratory method of incubating a sediment mesocosm, varying the overlying oxygen concentration, and measuring the reduced species with sediment depth through incubation time via the use of microelectrodes was shown to be appropriate for studying the sediment porewater response to episodic hypoxia.
CHAPTER III
CORPUS CHRISTI BAY, TX

Introduction

Hypoxia would not be expected in a shallow and windy bay such as Corpus Christi Bay, TX, since, it usually occurs in deeper waters coinciding with water column stratification (e.g. Mississippi River Bight). However, Corpus Christi Bay is of particular interest because in the summer months of July and August, when salinity stratification is greatest and temperature is highest, hypoxia does indeed occur (Ritter and Montagna, 1999). According to Ritter and Montagna (1999), hypoxia persisted for over three weeks in the summers of 1988 - 1996. The southeastern corner of the Bay exhibited chronic hypoxia. This region of the Bay may be more susceptible to hypoxia because this area has the least amount of water movement and is subject to stagnation because it is on the lee side of a barrier island. Morehead et al. (2002) assigned different locations in this region of the Bay a hypoxia likelihood index (HLI). “This index combines chemical measurements into a single value that creates a univariate index from multivariate data” (Morehead et al., 2002). This index was made over a seven-year study from 1994 – 2001, where the frequency of the hypoxic occurrence for a site was also plotted (Figure 3.1). The stations chosen for study in Corpus Christi Bay are based upon the results of the previous observations. The three stations chosen were station numbers 12, 10 and 24 as shown in Figure 3.1 and were renamed to CCB1,
Fig. 3.1. The hypoxic frequency patterns for stations in Corpus Christi Bay (based on Morehead et al., 2002). White = Well–mixed, no salinity or D.O. stratification. Dotted = Well–mixed, no salinity stratification, but D.O. decreases at depth. Black = Highly stratified with halocline and a decline in D.O.
CCB2, and CCB3, respectively. These stations were chosen randomly among those with a high likelihood of becoming hypoxic. Ritter and Montagna (1999) reported that the macroinfaunal community biomass was reduced by 1200% and abundance and diversity decreased by 500%, during the hypoxic events. Polychaete and oligochaete populations tolerated low oxygen conditions better than other infauna at these stations.

Past research has not included the sediment biogeochemistry and associated redox chemistry. This study describes both field observations and microcosm experimental results for major sediment biogeochemical parameters that are affected during a hypoxic event to help better understand this phenomenon. The expected biogeochemical behavior in the sediments in response to reduced oxygen condition is described in Figure 3.2. The purpose of this study was to test the conceptual model. At any fixed depth increment in a closed system, the model illustrates that, during oxic conditions, dissolved porewater concentrations of reduced species should be low, while during hypoxic periods concentrations should be elevated.

**Methodology**

**Study Location**

Corpus Christi Bay (Figure 3.3), the primary bay of the Nueces Estuary, is 500 km² in area with an average depth of 2 m and salinity of 14.8-31 (Montagna et al., 1996). The volume of the estuary is 1 km³, with a yearly freshwater inflow of 30 m³ s⁻¹, rainfall of 74 cm yr⁻¹, and evaporation of 151 cm yr⁻¹ (Morehead et al., 2002). The residence time is long at 5.5 month (Ritter and Montagna, 1999), which may promote
Fig. 3.2. Conceptual model of the ideal behavior of dissolved constituents in response to overlying water oxygen concentrations.
Fig. 3.3. A map of Corpus Christi Bay, TX. Solid circles represent the three chosen study sites in this research (based on Ritter and Montagna, 1999).
hypoxia, where the longer time a parcel of water is in the estuary (bottom waters) the more oxygen is consumed from it and the less quickly it becomes replenished. The maximal macrofaunal benthos abundance is 72 individuals m\(^{-2}\) (Montagna et al., 1996). Average monthly wind speeds range from 17 km h\(^{-1}\) to 28 km h\(^{-1}\) and are mainly out of the southeast direction (Morehead et al., 2002). Samples were taken during a 5 week period in Mid-July to Mid-August in the summer of 2002 and during a return in April of 2003.

**Sample Recovery**

Sediment cores were pushed-cored by SCUBA divers with care to minimize disturbance during the collection activities. Then they were promptly covered and clasped with fitted tops and immediately taken to the laboratory for further analyses. Replicates, controls, and all supplementary cores were retrieved within 1 m of one another or as close as otherwise possible. Cores that were taken from each site included two (one experimental, one control) 16 cm diameter, 40 cm-long Plexiglas incubation cores for microelectrode measurements, a 2 cm diameter, 30 cm-long Plexiglas sulfate reduction core with injection ports every 2 cm, a 7 cm diameter, 40 cm-long Plexiglas core for pore water squeezing and sediment cake preservation for dissolved inorganic carbon (DIC), sulfate to chloride (SO\(_4^{2-}/\text{Cl}^{2-}\)), total reduced sulfides (TRS), and reactive iron and manganese analyses, a 7 cm diameter, 40 cm-long Plexiglas core for microelectrode monitoring, a 7 cm diameter, 40 cm-long Plexiglas core for porosity
and grain size, and an 8 cm diameter, 20 cm - long Plexiglas core for sediment oxygen demand and nitrogen fluxes (Figure 3.4).

**Microelectrode Measurements**

Electrode measurements were made in the same manner as mentioned in Chapter II. However, after the electrode temporal profiling was terminated, the 16 cm diameter incubation cores were subcored for the $^{35}$S incubations, porewaters, and sediment cake samples in the same manner as mentioned below. Each site was monitored with repeat microelectrode core collection to assess heterogeneity and to compare the laboratory – to - natural setting; three profiles were made in these cores, at the same time, by “multiplexing” with three separate electrodes placed at the same depth. Control cores were aerated throughout the experiment entirety. Depth profiles were integrated for each measured species, only where the species was detected, to provide a single value for each time increment.

**Porewater and Solid Phase Analyses**

The sulfate reduction cores were collected, capped, and taken to the laboratory for injection of 10 µci / injection port Na$_2$$^{35}$SO$_4$, then incubated for 24 hours and frozen after the methods of Jørgenson (1978). The sulfate reduction rates were determined following the laboratory distillation and separation process using the boiling Cr (II) + acid method of (Canfield et al., 1986). Once the separation of the radiolabeled sulfide and sulfate fractions was made, the samples were then diluted with zinc acetate and
Fig. 3.4. Schematic of core collection and associated measurements.
measured on a LKB Wallack 1219 Rackbeta scintillation counter. Where, the ratio of the sulfide ($H_2^{35}S$) to sulfate ($^{35}SO_4^{2-}$) fractions was calculated. The “squeezer” core was taken to the laboratory, sliced in 2 cm sections under a nitrogen environment to prevent oxidation, and put on the squeezer rack where the sediments were pressurized with $N_2$. Pore waters for DIC and $SO_4/Cl$ were collected with 10 - ml syringes and refrigerated in glass vials with septum. Dissolved inorganic carbon was analyzed using a UIC carbon dioxide coulometer (Dickson and Goyet, 1994). Sulfate and chloride measurements were made on a Dionex ion chromatograph. The sediment cake samples for solid phase chemical analysis of reactive iron and manganese and total reduced sulfide (TRS) were immediately frozen. The citrate dithionate and cold HCl techniques (Raiswell et al., 1994) for reactive iron and manganese were utilized, where flame atomic absorption (Perkin Elmer 3110) was used to determine concentrations. The boiling Cr (II) + acid method for TRS analysis (Canfield et al., 1986) and the Cline (1969) $H_2S$ analysis method (Varian DMS 100 spectrophotometer) were used for sulfide extraction and detection. Organic carbon at each site was measured in the top 2 cm of sediment with a UIC carbon furnace and coulometer. The archive core was frozen immediately and the porosity/grain size core was left at room temperature for later analyses in the laboratory. Porosity measurements were made by comparing the wet and dry weights of sectioned, 2 cm sediment samples. Grain size analysis was done by sieving the 2 cm sections in the following chosen size intervals: 420 – 355 µm, 355 - 225 µm, 225 - 125 µm, 125 - 60 µm, and < 60 µm. Sediment oxygen demand was made by incubating the collected cores following the
method of Lavrentyev et al. (2000) where measurement of the O₂ to Ar ratio in the collected pore water samples was made via mass spectrometry following the method of Kana et al. (1994). During field collection, dissolved oxygen, salinity, and temperature for surface and bottom waters were recorded using a YSI meter.

**Results**

**Monitoring Field Study**

**2002 Field Conditions**

Since wind speed and direction can be important in mixing events in shallow estuaries, they are reported in Figure 3.5. The wind speed fluctuated from 0 – 11 m s⁻¹ (0 – 40 m hr⁻¹), but averaged 6 m s⁻¹ (21.6 m hr⁻¹) during the duration of the study. The wind direction also varied, but generally it was 150° – 200° from the North. Although the wind can be variable, it did not seem to have a large impact on the already hypoxic region of Corpus Christi Bay; however, it is likely that a major storm event could cause sufficient mixing to remove the hypoxic condition (Rabalais et al., 1994). During field activities the surface and bottom water temperature, salinity, and dissolved oxygen concentrations were measured using a YSI meter. The measured values are reported in Table 3.1. All sites showed relatively little difference in surface and bottom water temperatures (0.5°), but each had stratification with salinity (stratification index, σ, as great as 16) and oxygen (bottom < 2.5 mg L⁻¹, surface > 4.8 mg L⁻¹). At all sites, the surface was much less saline than the bottom values with both values becoming more
Fig. 3.5. a) Wind speed and b) direction during the course of field study (Taken from Texas A&M Corpus Christi website, http://dco.cbi.tamu.edu).
TABLE 3.1. Corpus Christi monitored sites location, temperature, salinity, and dissolved oxygen.

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<th>Long.</th>
<th>T (°C)</th>
<th>T (°C)</th>
<th>Sal.</th>
<th>Sal.</th>
<th>O₂ (mg L⁻¹)</th>
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</tbody>
</table>
fresh through the course of the study, indicating that runoff from earlier spring rains, were reaching the estuary. Previous research from Ritter and Montagna (1999) showed that hypoxia in the Bay correlated with the halocline, where the thickness of the bottom water hypoxia approached 1 m above the bottom. The bottom water oxygen at all stations was considered hypoxic at < 2.5 mg L\(^{-1}\) throughout the duration of study. Over half of the measured dissolved oxygen was less than 0.3 mg L\(^{-1}\), indicating the severity of the hypoxia. Previous research from Ritter and Montagna (1999) determined that the hypoxic oxygen values for Corpus Christi Bay should be considered any value < 3 mg L\(^{-1}\), further illustrating that these conditions were indeed severe for this estuary. Since the salinity stratification was pronounced at all stations, with hypoxic conditions reigning, bottom water dissolved oxygen was plotted against \(\sigma\), the salinity stratification index (Figure 3.6 a). Generally, as the stratification index became greater, the dissolved oxygen in the bottom water declined at all stations. Bottom water temperature, salinity, and oxygen are also plotted with time (Figure 3.6 b - c). Although the bottom waters were hypoxic and the surface waters had higher concentrations of oxygen (> 4.8 mg L\(^{-1}\)), for these temperature and salinity conditions they were not saturated with respect to oxygen (\(~ 6.24\) mg L\(^{-1}\)).

2002 Microelectrode Results

Measured porewater sulfide (H\(_2\)S) and iron (Fe\(^{2+}\)) profiles were integrated from 0 - 50 mm and averaged from three, separate, simultaneously taken profiles. This depth
Fig. 3.6. a). Bottom water dissolved oxygen vs. sigma for all stations. b). Bottom water temperature values through the study duration at all stations. c). Bottom water dissolved oxygen values through the study duration at all stations. d). Bottom salinity values through the study period at all stations.
was chosen because much of the chemical redox variability occurs in this region (refer to Chapter II). At stations CCB1 and CCB2 integrated sulfide values were initially low (<1 mmol m²), but as the study time progressed, they increased to a steady value (refer to Table 3.2). The integrated iron values at these stations were opposite to that of the sulfide; they were initially high and with time steadily decreased. At station CCB3, sulfide was continuously high while iron was low.

The general trend between the dissolved sulfide and iron chemistry showed an inverse relationship (Fig. 3.7) for all field results, however the Pearson’s correlation was not significant (p > 0.05). Furthermore, the same trend was seen with dissolved oxygen in the overlying waters and dissolved iron and sulfide in the porewaters, but this too was not significant, p > 0.05 (Fig. 3.8).

<table>
<thead>
<tr>
<th>TABLE 3.2. Corpus Christi “monitored” sediment porewater Fe²⁺ and ΣH₂S integrated (mmol m⁻²) concentrations with collection time.</th>
</tr>
</thead>
<tbody>
<tr>
<td>(hours)</td>
</tr>
<tr>
<td>---------</td>
</tr>
<tr>
<td>CCB1</td>
</tr>
<tr>
<td>ΣH₂S</td>
</tr>
<tr>
<td>Fe²⁺</td>
</tr>
<tr>
<td>CCB2</td>
</tr>
<tr>
<td>ΣH₂S</td>
</tr>
<tr>
<td>Fe²⁺</td>
</tr>
<tr>
<td>CCB3</td>
</tr>
<tr>
<td>ΣH₂S</td>
</tr>
<tr>
<td>Fe²⁺</td>
</tr>
</tbody>
</table>
Fig. 3.7. Monitoring cores integrated Fe$^{2+}$ and H$_2$S (mmol m$^{-2}$) for all stations through the field study duration.

Fig. 3.8. Monitoring cores integrated Fe$^{2+}$ (mmol m$^{-2}$) and O$_2$ (µM) for all stations throughout the field study duration.
Experimental Laboratory Study

Visual Observations

A light brown surface sediment was present during aeration and decreased in thickness during hypoxic conditions ~ 2 mm to ~ 0.5 mm. During hypoxia, the benthic biota such as the polychaete in Figure 3.9 would migrate into the overlying water to reach oxygenated water. With time, the burrows were abandoned, and no evidence of bioturbation was observed. The sediments also changed from grey - brown to black with decreased oxygen levels. These sediments appeared to have many (unquantified) worm holes and potential bioturbators but, by the end, inactivity by these was noted.

Microelectrode Study

Measured porewater total dissolved sulfide ($\Sigma H_2S$) and iron ($\text{Fe}^{2+}$) profiles were integrated from 0 - 50 mm. This depth was chosen because much of the chemical redox variability occurs in this region (refer to Chapter II). At stations CCB1 and CCB2 integrated sulfide values were initially low (< 9 mmol m$^{-2}$), but as the study time progressed, they increased to a steady value (refer to Table 3.3). At all three stations dissolved sulfide was released into the overlying waters at approximately 35 µM, 32 µM, and 40 µM concentrations for CCB1, CCB2, and CCB3, respectfully (see Appendix). Iron was also released into the overlying waters at an order of magnitude greater at approximately 1000 µM, 2500 µM, and 600 µM (see Appendix). Iron in the porewaters was elevated initially and with time steadily decreased. At station CCB3, sulfide began
Fig. 3.9. Photographs from laboratory cores during anoxia (left) re-oxic (right) conditions. They show oxidated sediment, bioturbators, and anoxic sediment.
TABLE 3.3. Corpus Christi sediment porewater Fe$^{2+}$ and H$_2$S integrated concentrations (mmol m$^{-2}$) and overlying O$_2$ concentrations (µM) with experiment time.

<table>
<thead>
<tr>
<th>(hours)</th>
<th>T1</th>
<th>T2</th>
<th>T3</th>
<th>T4</th>
<th>T5</th>
<th>T6</th>
<th>T7</th>
<th>T8</th>
<th>T9</th>
<th>T10</th>
<th>T11</th>
<th>T12</th>
</tr>
</thead>
<tbody>
<tr>
<td>CCB1</td>
<td>0</td>
<td>42.5</td>
<td>60.5</td>
<td>115</td>
<td>137</td>
<td>158.5</td>
<td>238</td>
<td>287.5</td>
<td>329.5</td>
<td>386.5</td>
<td>456.5</td>
<td>497</td>
</tr>
<tr>
<td>O$_2$</td>
<td>145.87</td>
<td>12.97</td>
<td>0.00</td>
<td>5.34</td>
<td>0.00</td>
<td>232.24</td>
<td>----</td>
<td>316.22</td>
<td>213.51</td>
<td>229.57</td>
<td>316.22</td>
<td>121.40</td>
</tr>
<tr>
<td>ΣH$_2$S</td>
<td>8.41</td>
<td>16.71</td>
<td>13.53</td>
<td>11.56</td>
<td>13.26</td>
<td>23.21</td>
<td>----</td>
<td>18.77</td>
<td>22.60</td>
<td>17.17</td>
<td>13.76</td>
<td>21.84</td>
</tr>
<tr>
<td>Fe$^{2+}$</td>
<td>13.46</td>
<td>53.59</td>
<td>51.09</td>
<td>40.24</td>
<td>13.71</td>
<td>11.95</td>
<td>0</td>
<td>8.20</td>
<td>0.56</td>
<td>2.41</td>
<td>0.54</td>
<td>10.40</td>
</tr>
<tr>
<td>CCB2</td>
<td>0</td>
<td>41.5</td>
<td>174.5</td>
<td>198</td>
<td>238</td>
<td>295</td>
<td>365</td>
<td>454</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>O$_2$</td>
<td>113.76</td>
<td>257.89</td>
<td>241.35</td>
<td>209.02</td>
<td>198.65</td>
<td>180.55</td>
<td>198.65</td>
<td>180.26</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>ΣH$_2$S</td>
<td>0</td>
<td>2.06</td>
<td>13.52</td>
<td>5.21</td>
<td>5.02</td>
<td>1.52</td>
<td>0</td>
<td>1.66</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fe$^{2+}$</td>
<td>51.22</td>
<td>62.59</td>
<td>100.46</td>
<td>97.83</td>
<td>0</td>
<td>0.54</td>
<td>0</td>
<td>69.33</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CCB3</td>
<td>0</td>
<td>48.5</td>
<td>88.5</td>
<td>144</td>
<td>216</td>
<td>255</td>
<td>351</td>
<td>398</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>O$_2$</td>
<td>241.35</td>
<td>15.79</td>
<td>0.00</td>
<td>0.00</td>
<td>212.84</td>
<td>244.36</td>
<td>105.84</td>
<td>93.82</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>ΣH$_2$S</td>
<td>24.22</td>
<td>10.45</td>
<td>12.90</td>
<td>27.72</td>
<td>22.05</td>
<td>12.74</td>
<td>14.79</td>
<td>12.61</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fe$^{2+}$</td>
<td>0</td>
<td>61.56</td>
<td>32.64</td>
<td>10.14</td>
<td>55.69</td>
<td>34.63</td>
<td>41.11</td>
<td>21.50</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

elevated and then decreased, while iron began at undetectable concentrations and then increased to detectable levels, during anoxic stage 1. This may, however, be a result of initial aeration. During re-aeration, iron generally decreased at all sites, while sulfide had a delayed, moderate decrease. During stage 2 anoxia, both iron and sulfide increased except at CCB3. Figure 3.10 depicts the core response at each station more vividly. In a manner similar to the field results, the low oxygen laboratory results generally showed an opposing relationship between the porewaters dissolved sulfide and iron, however the Person’s correlation was not significant (p > 0.05). The same general inverse relationship between dissolved oxygen in the overlying waters and dissolved iron
Fig. 3.10. Dissolved iron (Fe\textsuperscript{2+}) and sulfide (ΣH\textsubscript{2}S) response to manipulated oxygen in the overlying waters with time in experimental cores.
Fig. 3.11. Experimental cores integrated Fe$^{2+}$ and $\Sigma H_2S$ (mmol m$^{-2}$) for all stations through the experiment duration.

Fig. 3.12. Experimental cores integrated Fe$^{2+}$ (mmol m$^{-2}$) and O$_2$ (µM) for all stations through the experiment duration.
in the porewaters was also noted, but without any statistical significance (p > 0.05), similar to field results (Fig. 3.11 and Fig. 3.12).

Control Cores

Laboratory control cores had oxygen values in the overlying waters (10 mm above sediment – water interface) > 200 µM. Within the sediment, iron concentrations were about half that of experimental cores (< 700 µM) and sulfide values were < 40 µM at all depths (Figure 3.13). Little increase in concentrations were noted with time of either dissolved sulfide or iron and overlying water remained oxic in control cores throughout the entirety of the experiment. No measurable release of either sulfide or iron into the overlying waters was found for any control core.

Porewater and Solid Phase Study

Solid phase (total reactive sulfides, reactive iron and manganese, sulfate reduction rates, porosity and grain size) and porewater (sulfate to chloride) measurements were made on initial field conditions and after the termination of the control and experimental cores. For all results, there is little observable difference between initially collected cores and final experimental cores. Average depth values for initial, final, and control cores show little change in any of the analyzed constituents (Table 3.5).
Fig. 3.13. Experimental control cores microelectrode profiles averaged concentrations (µM) for O₂, Fe²⁺, and ΣH₂S.
Total reduced sulfide increased with depth at all stations and ranged from 10 – 70 µmol g⁻¹ sediment (see appendix). CCB3 and CCB1 showed a decline in TRS after experimentation which was most likely due to sub-coring after laboratory aeration however, without destroying the cores and terminating the experiment, TRS measurements after non-aeration (induced hypoxia) could not be sampled. TRS values were half of that found in the Mississippi River Bight (Morse et al., 1999). There was < 0.5 µmol g⁻¹ sediment of reactive manganese at all sites, supporting electrode measurements of no detected dissolved manganese. Reactive iron was present at all stations and the amount of HCl extractable iron was greater than the amount of citrate dithionate extractable iron. The more aggressive HCl technique extracted 15 – 40 µmol Fe g⁻¹ sediment, while the citrate dithionate technique extracted 2 – 10 µmol Fe g⁻¹ sediment. Reactive Fe was more than an order of magnitude less than other coastal areas in the Gulf of Mexico (Morse et al., 2002) and was most likely due to decreased supply of iron oxides from river input. The grain size analyses indicate that the sediments were sandy with the majority of the grains in the 125 – 212 µm size fraction. The sulfate to chloride porewater results show a general trend for all stations had experienced sulfate reduction and depleted sulfate (< 28 mM). Further laboratory hypoxia decreased dissolved sulfate even further. Sulfate reduction rates (SRR) were highest at CCB3 along with the benthic oxygen demand and nitrate fluxes (Table 3.4). Organic carbon (%) was lowest at CCB1, moderate at CCB2, and highest at CCB3.

Carbon remineralization values were calculated from sulfate reduction rates and benthic oxygen demand for each station (Table 3.4). Values were greater than 300 mg C
m$^{-2}$ day$^{-1}$ for remineralization rates calculated from benthic oxygen demand and more than 4 – times greater for those calculated from sulfate reduction rates. This discrepancy is due to the very low O$_2$ concentration in the bottom water (Morse and Rowe, 1999; Rowe et al., 2002) depressing the benthic oxygen demand. Carbon remineralization values calculated from sulfate reduction were more than double Mississippi River Bight rates which ranged from 195 - 414 C m$^{-2}$ day$^{-1}$ (Rowe et al., 1999), revealing the benthic metabolic activity in the Bay.

Using the benthic oxygen demand of ~ 1 mmol O$_2$ m$^{-2}$ hr$^{-1}$ and letting the bottom water hypoxic thickness to equal 1 m (Ritter and Montagna, 1999), the onset of hypoxia in a closed system for this Bay was approximately < 5.5 days. This indicated that in the southeastern region of the Bay, where mixing was minimal and the water column was shallow, the sediments alone could have caused the onset of the hypoxic event in a relatively short time period.

Furthermore, the relationship between the integrated sulfate reduction rate and the benthic oxygen demand can be made. Assuming, the reduction of organic carbon involves 2:1 ratio of sulfate to oxygen, the percent of oxygen needed to oxidize the amount of sulfide present in the porewaters can be calculated (Table 3.4). At all stations this was > 100%, because each station was an oxygen depleted environment, where the amount of sulfide was greater than the amount of free oxygen and therefore all of the available oxygen was used in oxidizing the sulfide to sulfate. Therefore, the reduced
### TABLE 3.4. Sediment organic carbon, benthic oxygen demand, sulfate reduction rates, organic carbon remineralization rates, and the maximum fraction of oxidized sulfate (OC = organic carbon; BOD = benthic oxygen demand; OCRR = organic carbon remineralization rates; SRR = sulfate reduction rate).

<table>
<thead>
<tr>
<th>OC</th>
<th>BOD</th>
<th>OCRR</th>
<th>SRR</th>
<th>OCRR</th>
<th>BOD*</th>
<th>Max. Fraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>%</td>
<td>(mmol O₂ m⁻² hr⁻¹)</td>
<td>(mg C m⁻² day⁻¹)</td>
<td>m⁻² hr⁻¹</td>
<td>day⁻¹</td>
<td>m⁻² hr⁻¹</td>
<td>(SRR*2)</td>
</tr>
<tr>
<td>SITE CCB1</td>
<td>0.22</td>
<td>0.420</td>
<td>302.70</td>
<td>0.96</td>
<td>2767.56</td>
<td>1.92</td>
</tr>
<tr>
<td>SITE CCB2</td>
<td>0.51</td>
<td>0.711</td>
<td>512.43</td>
<td>1.20</td>
<td>3747.74</td>
<td>2.60</td>
</tr>
<tr>
<td>SITE CCB3</td>
<td>0.79</td>
<td>0.765</td>
<td>551.35</td>
<td>2.49</td>
<td>7192.79</td>
<td>4.99</td>
</tr>
</tbody>
</table>

### TABLE 3.5. Average porewater (dissolved sulfate) and solid phase (total reduced sulfide (TRS) and reactive iron – citrate dithionate (CD) and cold HCl) results, for all sediment depths, at Corpus Christi Bay (see Appendix for values at each depth).

<table>
<thead>
<tr>
<th>TRS</th>
<th>SO₄²⁻</th>
<th>Fe – CD</th>
<th>Fe – HCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>(µmol g⁻¹ sed.)</td>
<td>mM</td>
<td>(µmol g⁻¹ sed.)</td>
<td>(µmol g⁻¹ sed.)</td>
</tr>
<tr>
<td>CCB1 initial</td>
<td>20.87</td>
<td>25.37</td>
<td>2.62</td>
</tr>
<tr>
<td>CCB1 end</td>
<td>16.11</td>
<td>21.83</td>
<td>6.40</td>
</tr>
<tr>
<td>CCB1 control</td>
<td>14.29</td>
<td>26.57</td>
<td>4.93</td>
</tr>
<tr>
<td>CCB2 initial</td>
<td>21.33</td>
<td>22.02</td>
<td>4.22</td>
</tr>
<tr>
<td>CCB2 end</td>
<td>21.46</td>
<td>23.30</td>
<td>7.95</td>
</tr>
<tr>
<td>CCB2 control</td>
<td>18.84</td>
<td>23.29</td>
<td>7.61</td>
</tr>
<tr>
<td>CCB3 initial</td>
<td>57.60</td>
<td>25.41</td>
<td>3.96</td>
</tr>
<tr>
<td>CCB3 end</td>
<td>33.86</td>
<td>23.96</td>
<td>3.86</td>
</tr>
<tr>
<td>CCB3 control</td>
<td>30.72</td>
<td>25.51</td>
<td>4.04</td>
</tr>
</tbody>
</table>
components acted as an indirect biological source in abiotic consumption of oxygen (even though they are biotically produced). The sulfide that was not oxidized remained to either complex to form iron sulfide minerals or to diffuse into the overlying waters.

Using the initial sulfate reduction rates (assumed SRR constant) and microelectrode porewater sulfide values, a determination of the distribution of sulfide in the porewaters versus solid phase was made for each station during hypoxia. Equations 12 - 13 were used to determine this ratio, where \( R_t = \text{SRR} \) and \( C_{\text{ideal}} = \text{integrated } \Sigma \text{H}_2\text{S.} \)

\[
C_{\text{ideal}} = C_i + R_t
\]  

(12)

\[
C_{tx} - C_{ix} = + \text{ or -}
\]

If negative, then \( S^2^- \) missing (precipitated, transported, or oxidized)  
If positive, then \( S^2^- \) remains in the porewaters

This calculation assumed a closed system and results illustrated the distribution ratio of the dissolved sulfide pool with time or sulfide production. For each station the results were positive (CCB1 = + 11.33, CCB2 = + 1.15, CCB3 = + 2.01), and illustrated that the sulfide produced remained in the porewaters and did not become completely precipitated or lost to transport. Further, this porewater build-up of \( \Sigma \text{H}_2\text{S} \) was due to the lack of oxidation since the conditions were hypoxic.
2003 Field Monitoring During Norm - Oxia

During the following spring, a return to each of the three stations was made. Field cores were collected and immediately profiled in the laboratory. This enabled a seasonal norm – oxic comparison for the previous hypoxic study. Results showed that all sites exhibited bottom water norm – oxia with dissolved oxygen greater than 3 mg L\(^{-1}\) (Table 3.6). Further, no iron or sulfide was detected in the overlying water. Salinity values were approximately 28, with little – to – no stratification between surface and bottom water values, and temperatures were constant at \(\sim 22 \, ^\circ\text{C}\). In – situ surface waters and laboratory microcosm measurements revealed that oxygen was saturated (\(\sim 6.0 \, \text{mg L}^{-1}\)) in the overlying waters. Dissolved iron and sulfide were present in the porewaters at all stations, however, at concentrations about half that of those during the summer study (Fe\(^{2+}\) < 408.84 \(\mu\text{M}\) and \(\Sigma\text{H}_2\text{S} < 32.72 \, \mu\text{M}\)). Oxygen penetrated to at least 3 mm into the sediment and sulfide was not detectable until below 10 mm. The integrated iron concentration was greatest at station 3, while the integrated sulfide concentration was the least at this station (Fig. 3.14). This supported the previously observed trend that when iron was high, sulfide was low and visa versa. Further the general trend between sulfide and iron and was inversed but without statistical significance (\(p > 0.05\)), thus the reduced species, during the norm – oxic season, seemed to follow observed laboratory trends.
TABLE 3.6. 2003 field (top) and laboratory (bottom) results.

<table>
<thead>
<tr>
<th>Site</th>
<th>Surface T (˚C)</th>
<th>Bottom T (˚C)</th>
<th>Surface Sal.</th>
<th>Bottom Sal.</th>
<th>Surface O2 (mg L⁻¹)</th>
<th>Bottom O2 (mg L⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CCB1</td>
<td>22.0</td>
<td>21.9</td>
<td>27.8</td>
<td>28.4</td>
<td>5.97</td>
<td>4.00</td>
</tr>
<tr>
<td>CCB2</td>
<td>21.9</td>
<td>21.8</td>
<td>28.7</td>
<td>28.7</td>
<td>6.29</td>
<td>6.14</td>
</tr>
<tr>
<td>CCB3</td>
<td>21.9</td>
<td>21.9</td>
<td>28.7</td>
<td>28.6</td>
<td>6.40</td>
<td>6.15</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Site</th>
<th>Overlying O2 (µM)</th>
<th>Fe²⁺ mmol m⁻²</th>
<th>ΣH₂S mmol m⁻²</th>
</tr>
</thead>
<tbody>
<tr>
<td>CCB1</td>
<td>745.82</td>
<td>4.89</td>
<td>2.03</td>
</tr>
<tr>
<td>CCB2</td>
<td>651.61</td>
<td>3.30</td>
<td>1.67</td>
</tr>
<tr>
<td>CCB3</td>
<td>777.07</td>
<td>9.68</td>
<td>0.16</td>
</tr>
</tbody>
</table>

Fig. 3.14. (Left) Averaged overlying water oxygen vs. integrated sediment iron for each site and (Right) averaged integrated sulfide vs. iron for each site in 2003.
Iron Sulfide Saturation States

The equilibrium between the dissolved and solid phases can be a major factor in the uptake and release of iron and sulfide. Usually > 98% of the sulfide is oxidized, but under hypoxic / anoxic conditions sulfide can build in both the porewaters and potentially more importantly in the solid phase as iron sulfides. Iron sulfide pools normally act as a buffer binding the excesses of sulfide (and iron) as FeS and FeS$_2$, preventing them from leaching into the overlying waters (Jørgenson, 1980). Canfield et al. (1992) discussed that sulfide should not accumulate into solution until after the consumption of the iron oxyhydroxides are made through iron sulfide formation. Once this iron pool is depleted, the less reactive iron phases react with sulfide at rates much slower than sulfide production (Canfield et al., 1992). Therefore, if the most reactive iron phases are no longer kinetically available to bind with the sulfide, then this buffer is no longer present and most of the dissolved sulfides will exclusively be in the porewater phase, where then diffusion into the overlying waters is possible.

For each site and for each treatment, the iron sulfide saturation state was calculated from electrode measurements for $\Sigma$H$_2$S and Fe$^{2+}$ (Fig. 3.15). It was found that all sites and all treatments were supersaturated with respect to mackinawite (FeS$_{0.9}$). However, controls and 2003 field monitoring cores during norm – oxic regimes showed values closer to equilibrium than laboratory experiments and 2002 hypoxic field monitoring cores. This was most likely due to the decreased concentrations of both iron and sulfide during oxic conditions and their less frequent coexistence in the sediment porewaters.
Fig. 3.15. Mackinawite (Fe$_{0.9}$) equilibria for all Corpus Christi sites and treatments (modified from Morse, unpublished).
**Statistical Analyses**

Data were analyzed using the SPSS© statistical package and tests preformed included Pearson’s and Spearman’s correlations and a multivariate analysis of variance (MANOVA). Data were first tested for normality (Kolmogorov – Smimov) and homogeneity of variance (Levene). Most data were normal and attempted transformations did not improve normality for the few that were not (p < 0.05). Not all data were homogeneous so therefore, Tamhane’s test for multiple comparisons was used (p < 0.05). Data were equally varied according to Box’s test (p < 0.05) and independent. Boxplots for both iron and sulfide are shown in Figures 3.16 – 3.17, outliers were not removed from the data set.

The correlation between iron (Fe$^{2+}$) and sulfide (ΣH$_2$S) was tested. Correlation values of +1 represent perfect positive correlation, values of 0 represent no correlation, and values of −1 represent perfect negative correlation. Both the nonparametric (Spearman) and parametric (Pearson) correlation analyses were conducted however no discrepancies between the tests were seen. According to the test, ΣH$_2$S and Fe$^{2+}$ had no correlation (p > 0.05) and the overlying water O$_2$ had no correlation (p > 0.05) with the porewater ΣH$_2$S and Fe$^{2+}$. However, general trends in the data indicate that sulfide and iron respond to changes in the overlying water.

The analysis of variance was conducted by arranging the iron and sulfide data into the four treatments of: laboratory non-aeration (1), field 2002 (2), field 2003 (3), and laboratory aeration (4). Further, data were arranged in respect to the sites (CCB1, CCB2, and CCB3) from which each of the cores was recovered. Results indicated the
Fig. 3.16. Sulfide (left) and iron (right) boxplots for the treatment of site.

Fig. 3.17. Sulfide (left) and iron (right) boxplots for the treatment of experiment.
H_0, the factor level means were equal, was rejected (p < 0.05) for the multiple comparisons test as shown below (a continuous underline indicates that means are not significantly different).

<table>
<thead>
<tr>
<th>Factor: ΣH_2S / Treatment: Site</th>
<th>Factor: Fe^{2+} / Treatment: Site</th>
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</thead>
<tbody>
<tr>
<td>CCB1   CCB3   CCB2</td>
<td>CCB1   CCB2   CCB3</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Factor: ΣH_2S / Treatment: Experiment</th>
<th>Factor: Fe^{2+} / Treatment: Experiment</th>
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</thead>
<tbody>
<tr>
<td>1 2 4 3</td>
<td>1 2 4 3</td>
</tr>
</tbody>
</table>

These results most importantly illustrated that experiment 3 (field data from 2003 during bottom water norm – oxia) gave sulfide results that were significantly different from any of the other experiments. This was important to show that the sulfide conditions measured during the (summer) hypoxic conditions at Corpus Christi Bay did not exist yearly and the hypoxic conditions did indeed change the sediment biogeochemistry and the sulfide dynamics were indeed affected. There was likely no significant difference shown for experiment 4 (core aeration periods) and the other laboratory experiments because of the small change in the dissolved components during the switch in aeration. Further, the time length of aeration may have needed to be longer to obtain a significant difference in the dissolved constituents. Site CCB2 sulfide was significantly different than the other sites which may indicate a physical, chemical or
biological mechanism that separates these sites, allowing for different sulfide characteristics there.

**Discussion**

**2002 Field and Experimental Laboratory Studies**

**Microelectrode Study**

Throughout the 2002 summer study period, the study sites underwent seasonal hypoxia due to strong salinity stratification. Both field and laboratory observations gave trends indicating an inverse relationship between oxygen in the overlying water and dissolved porewater iron (Fe$^{2+}$) and sulfide (H$_2$S). This was probably due to the dissolved iron maximum occurring relatively close to the sediment – water interface and thus Fe$^{2+}$ was oxidized through its upward diffusion and the downward diffusion of O$_2$. This observation was supported by a similar finding by Kristainsen et al. (2002), where Fe$^{2+}$ fluxes across the sediment-water interface were inversely correlated to the concentration of O$_2$ in the overlying waters. Sulfide would also become oxidized through the same manner as iron, but because of its broader vertical distribution it would likely take longer for its integrated value to decline. The relationship between oxygen and iron and sulfide illustrates the model’s (Figure 3.2) fit for the chemical response to hypoxic conditions. With increases in oxygen there were decreases in the reduced components and visa versa. Further, both field and laboratory observations showed trends indicating an opposing relationship between dissolved integrated ΣH$_2$S and Fe$^{2+}$. 


In laboratory studies there was a consistent lower integrated concentration of Fe$^{2+}$ and a delayed decrease in integrated $\Sigma H_2S$ during re-oxidation. There was an increase in Fe$^{2+}$ during induced hypoxic periods and a “banking” of porewater $\Sigma H_2S$. This “banking” effect may be due to a lack of H$_2$S oxidation, an increase in metabolism of sulfate reducers, an upward diffusion for sulfide under the measured zone, and a decline in bioturbation. This could have impacts on the bottom water recovery from a hypoxic event. The stored sulfide will eventually be oxidized when bottom waters become replenished with oxygen and downward diffusion of O$_2$ occurs. Therefore sulfide would be an oxygen sink, consuming the available oxygen and delaying recovery. Similar findings in the Chesapeake Bay sediments (Roden and Tuttle, 1992) showed that sulfur cycling alone can maintain anoxia in subpycnocline waters during summer hypoxia in deep waters.

Control cores supported the validity of the experiment, undergoing continual oxic conditions, where sulfide and iron concentrations were significantly less than experimental cores and exhibited little change with experiment time.

*Porewater and Solid Phase Study*

Sediment analyses supported microelectrode measurements with the presence of reactive iron and reduced sulfide, with little difference between the initial, control, and final cores. This was most likely due to the stable nature of these pools and the small changes that may be seen during short-lived hypoxic conditions. The majority (300x) of the total sulfide was found in the solid phase component of the sediment and not in
the dissolved portion of the porewaters based on the TRS (~ 20 µmol g⁻¹) and ΣH₂S (~ 40 µM) values. Therefore, small changes over short time scales to the solid phase portion would not be very noticeable. Furthermore, although the porewater phase was a very small portion of the total sulfide pool, it was of crucial importance because it was the mobile phase, which can have the most adverse affect on the water chemistry and associated fauna.

The Mississippi River Bight (MSB) is an area that is similar in seasonal hypoxia to Corpus Christi Bay and a good site for comparison, especially since studies describing the benthic response under hypoxic conditions are limited. Results from the MSB (Morse and Rowe et al., 1999) showed higher total reduced sulfide values (104.2 µmol g⁻¹ sed.) than the results in this study, but sulfate reduction rates (0.67 mmol SO₄²⁻ m⁻² hr⁻¹), benthic oxygen demand (1.9 mmol O₂ m⁻² hr⁻¹), and organic carbon (0.47 %) values were in good agreement with the Corpus Christi Bay sediments (TRS = 14.29 – 57.60 µmol g⁻¹ sed.; SRR = 0.96 – 2.49 mmol SO₄²⁻ m⁻² hr⁻¹; BOD = 0.420 – 0.765 mmol O₂ m⁻² hr⁻¹; OC = 0.22 - 0.79 %). The findings showed the results of the present study were indeed indicative of a hypoxic region and the benthic biogeochemistry in seasonally hypoxic areas may be similar for the Gulf Coast.

The formation of FeS was the buffer for the Corpus Christi Bay sediments and the controlling factor preventing sulfide release. Mackinawite was supersaturated (Ω > 10) at all stations for both field and experimental cores. However, both ΣH₂S and Fe²⁺ were released into the overlying waters during induced hypoxic experimentation and field hypoxia, indicating that the buffering capacity of the formation of iron - sulfide
minerals was not sufficient to prevent the leaching of the two redox species. Where iron was released at concentrations an order of magnitude greater than sulfide and therefore may be a more important consumer of oxygen. The formation of FeS minerals required the presence of both iron and sulfide so where iron was not detectable, sulfide could easily leach out of the sediment and into the oxygen deprived overlying waters, where it would not be oxidized until further up into the water column. The formation of iron-sulfide minerals for control cores were closer to equilibrium ($\Omega = 1.01 – 6.04$) suggesting that norm-oxic conditions, decreased the formation of these minerals.

Seasonal changes in the FeS pool was also noted by Jørgenson (1980) in a Danish fjord, where peak concentrations of FeS were found when overlying oxygen concentrations were lowest. Increases in both pyrite formation and the acid soluble ferric iron were seen in the summer, where iron was an important barrier to the release of $\text{H}_2\text{S}$ into the anoxic bottom water (Jørgenson, 1980).

2003 Field Monitoring Study

Field results from the 2003 study suggested that the sediment redox, during seasonal norm-oxia, agreed with laboratory results. Dissolved integrated $\Sigma\text{H}_2\text{S}$ and $\text{Fe}^{2+}$ in the porewaters generally showed trends of an inverse relationship with each other and with oxygen. Oxygen concentrations in the overlying waters were saturated and both sulfide and iron concentrations were minimized in the porewaters and were not detectable until deeper sediment depths. This indicated that the downward diffusion of
oxygen allowed for oxidization of the two species, the aerobic respiration was present further downcore, and the metabolism of sulfate and iron reducers was lowered.

Previous research by Aller (1980) supported this seasonal change in the redox species depth profiles. Maximum concentrations of Fe\(^{2+}\) and Mn\(^{2+}\) decreased during the fall relative to those in the summer and the vertical extent of concentration peaks were broadened in the Long Island Sound sediments (Aller, 1980). This suggested that a combination of lowered production rates and relatively increased importance of biogenic transport during the winter was the cause for the changes (Aller, 1980). The decreased microbial activity was associated with lowered water temperatures and the transport effects of biogenic reworking and irrigation therefore dominated (Aller, 1980). The current research paralleled the previous work where during norm-oxic periods the concentrations of both Fe\(^{2+}\) and \(\Sigma H_2S\) were lowered due to seasonal effects as aforementioned.

The formation of iron-sulfide minerals was closer to equilibrium (\(\Omega = 3.0 - 9.23\)) during 2003 seasonal norm-oxic cores, this is most likely due to the lowered concentrations of reduced iron and sulfide during such regimes. Furthermore, \(\Sigma H_2S\) and Fe\(^{2+}\) were never released into the overlying waters, suggesting that the FeS buffering capacity was sufficient to prevent their release.

**Overview**

Sulfide and iron seemed to undergo different dynamics during a hypoxic episode at Corpus Christi Bay, suggesting that each of these carbon degradation bacterial
pathways were favored during certain phases of the hypoxic episode. Iron and sulfide results followed the idealized model shown in Figure 3.2, however, the need for more data during each treatment would be recommended. The Corpus Christi Bay sediments possessed the ability to prevent extenuating damage to the fauna by inhibiting sulfide release with the formation of iron sulfides for short hypoxic periods (< 200 hours). However, with time, this buffer capacity was exceeded and this fatal constituent was released into the overlying waters once the dissolved iron pools were priory completely released. Furthermore, the “banking” of sulfide in the porewaters can delay the recovery of the bottom waters from a hypoxic event because of consumption of oxygen during sulfide oxidation.

Conclusions

This study has indicated that the hypotheses were true where manipulation (decrease / increase) of overlying water O₂ concentrations caused an associated (upward / downward) migration of reduced iron and sulfide, laboratory experimental and field results agreed with respect to the iron and sulfide dynamics during a hypoxic event and during norm – oxia, and during early hypoxic conditions (< 200 hours), the formation of FeS minerals prevented sulfide from leaching into the overlying waters. But during induced experimental and field hypoxia, reduced iron and sulfide were released into the overlying waters at hypoxia > 200 hours. Further, the build up of dissolved Fe²⁺ and ΣH₂S was sufficient to delay recovery from the hypoxic event since the demand of the oxygen required to oxidize these species was greater than that available. During norm -
oxic conditions the concentrations of both species (Fe^{2+} and ΣH_{2}S) were significantly less.

This research has been preliminary in nature, but further studies like this need to be included in benthic - pelagic coupling models. Brief dramatic changes in the redox chemistry (Fe^{2+} and ΣH_{2}S) during hypoxic events, because of biogeochemical processes, show typical phase lags with associated changes in the overlying water. The leaching of redox species into the overlying waters contributed to the hypoxic event by the abiotic consumption of the available oxygen, prolonging its duration and possible damage to the benthic fauna and flora. Reduced iron may be as important, if not more important, to a hypoxic event as sulfide because of its diffusion into the overlying waters at concentrations an order of magnitude greater than sulfide. The iron chemistry illustrated that the preferred emphasis by researchers on sulfide chemistry during hypoxia may be misguided and more work on all of the redox reactive species needs to be conducted in order to accurately assess the impact of the sediment on hypoxia. This work has primarily shown that one cannot monitor the water without knowing the sediment biogeochemistry and its contribution to the system, especially in shallow – water areas where hypoxia persists.
CHAPTER V
OVERVIEW AND SUMMARY

Generalizations

A method has been exhibited where induced hypoxia and the corresponding redox parameters were measured in the sediments. Field and laboratory trends seem to be in general agreement. Dissolved iron (Fe$^{2+}$) may play a more important role in the sediment biogeochemistry than may have previously been thought during episodic hypoxia. Since it seemed to, in part, impact the diffusion of H$_2$S into the overlying waters and become leached into the overlying waters itself, at higher (order of magnitude) concentrations than sulfide. Dissolved sulfide (ΣH$_2$S) may have delayed effects on the system because of its tendency to “bank” in the sediment porewaters. The buffering of the dissolved H$_2$S is the result of equilibria between dissolved and solid phases of iron and sulfide. However, the buffer capacity at certain locales can vary, and therefore it is important to know the sediment biogeochemistry before the impact of hypoxia can be assessed at any one location. Thus, this has been a demonstration that without fully understanding the changes that occur within the sediment biogeochemistry, one can not assess the overall characteristics of the benthic – pelagic system. Further study needs to be made to completely understand the processes that occur during a hypoxic episode in Corpus Christi Bay and the associated redox parameters, which can have a significant impact on the system.
Future Study Recommendations

Some modifications to the experimental technique would include a more precise way to regulate \( O_2 \) concentrations to experimental cores (i.e. compressed \( O_2 \) gas or nitrogen stripping). For statistical strength, more replicates at each oxygen concentration level, with time, on multiple cores for each station is needed where an analysis of variance or a repeated measures analysis can be made to further determine a relationship with hypoxic versus normoxic regimes. Modifications in the experimental design are also needed where longer times lengths for oxygen treatments to obtain more data points per treatments are needed. If more replicates with time are performed, then the exact oxygen concentration that the sediment can tolerate before reduced species release can be found. Further, the time increments for both laboratory and field data need to coincide more precisely for statistical comparison purposes. A continuous flow system for each core would be a valued addition serving as the constant stirring mechanism (Miller–Way et al., 1994). Further the addition of benthic chambers to obtain the in situ sediment water interface fluxes for DIC, dissolved sulfate, and oxygen would be a valued measurement. A modeling approach where the collected data is integrated into a diagenetic model (Eldrige and Morse, 2000) and the implementation of a hypoxic model for the Texas Coast would be a goal for the future. Imbedded would be a model where the physical, biological, and chemical impacts of each study site are incorporated. This would be the only way to completely understand the system and the causes and effects of hypoxic episodes on the benthic biogeochemistry and associated mechanisms. Evidence for the effectiveness of such a model has already been seen in the similarities in the
benthic biogeochemistry between the Mississippi River Bight and Corpus Christi Bay and therefore it would be appropriate in describing at least two of the known hypoxic regions of the Gulf Coast.
REFERENCES


Jorgensen B.B. 1980. Seasonal oxygen depletion in the bottom waters of a Danish fjord and its effect on the benthic community. OIKOS 34: 68-76.


Morehead, S., Simanek, C. Montagna P.A. 2002. GIS database of hypoxia (low oxygen) conditions on Corpus Christi Bay. Coastal management program technical report. University of Texas Marine Institute, Port Aransas, TX.


Fig. A1. Porosity measurements for Corpus Christi Bay stations.

Legend

- % < 63 µm
- % 63 – 125 µm
- % 125 – 212 µm
- % 212 – 355 µm
- % 355 – 425 µm
Fig. A2. Reactive iron measurement for Corpus Christi Bay stations.

Fig. A3. Dissolved sulfate for Corpus Christi Bay Stations.
Fig. A4. Total reduced sulfide for Corpus Christi Stations.

Fig. A5. Corpus Christi Bay Station 1 iron and sulfide profiles (letters correspond with the first five times in Table 3.4).
Fig. A6. Corpus Christi Bay Station 2 iron and sulfide profiles (letters correspond with the first four times in Table 3.4).

Fig. A7. Corpus Christi Bay Station 3 iron and sulfide profiles (letters correspond with the first four times in Table 3.4).
TABLE A1. Sulfur solid phase and porewater analyses for both Corpus Christi Bay and the Mississippi River Bight, total reactive sulfide, porewater sulfate, and sulfate reduction rates.

<table>
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<tr>
<th>Station</th>
<th>Depth (cm)</th>
<th>TRS µmol g⁻¹</th>
<th>SO₄²⁻ mM</th>
<th>³⁵S mmol SO₄m⁻²day⁻¹ CCB1initial</th>
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CCB1_M2
Trode 1
144 hours

CCB1_M2
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398 Hours

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VITA

Karen S. Sell

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Bachelor of Science earned from Eckerd College, Saint Petersburg, FL
Major: Marine Science    Minor: Environmental Studies
Cumulative Grade Point Average: 3.61/4.0 Scale
Graduate Career Cumulative Grade Point Average: 3.80/4.0 Scale

PUBLICATIONS (6) / PRESENTATIONS (4) / ABSTRACTS (1):

- Sell K.S. and Morse J.W. 2003. Temporal influences of seasonal hypoxia on sediment biogeochemistry in coastal sediments. Presented at the Gulf Coast Estuarine Research Symposium, University of Texas Marine Science Institute, Port Aransas, TX.